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PATENT SPECIFICATION

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DRAWINGS ATTACHED.

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Int. Cl.:—C 22 b, 9/04 //B 22 c, d, C 22 c.

COMPLETE SPECIFICATION.

Refining Tower for Copper and Copper Alloys.

We, ASSOCIATED ELECTRICAL INDUSTRIES LIMITED, a British Company having its registered office at 33 Grosvenor Place, London, S.W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention is concerned with the refining of copper and copper alloys, and is particularly concerned with an apparatus and a procedure for accomplishing the almost complete removal of oxygen from copper and copper alloys when in the molten state.

The invention comprises a graphite refining tower which can be run under high vacuum and at high temperatures during the process of refining the metal. The tower is heated externally, by electrical means, and is made with longitudinal slots in the outer surface of the graphite, these slots being spaced equidistantly round the tower. The effect of these slots is to increase the heating efficiency and give smoother running of the refining process. The entire apparatus is enclosed in a fused silica envelope, within which the necessary high vacuum is created by known mechanical means. Heating is supplied from one or more electrical induction coils surrounding the silica envelope.

The theory of the refining process is based on the equation:—

$$Cu_2O + C \rightleftharpoons 2\,Cu + CO$$

where there is a positive free energy change which increases as the partial pressure of carbon monoxide is decreased and as the temperature is increased.

Oxygen is in solution in liquid copper during the refining process, and if oxygen is to be lost from the melt, oxygen atoms must diffuse to the surface and escape from the surface as gas. At the surface, copper atoms as vapour tend to combine with oxygen gas to form cuprous oxide molecules which then condense back into the surface of the molten copper. It is known that the more dilute the oxygen solution in molten copper the lower the dissociation pressure of oxygen. Thus in a plain vacuum, as the oxygen content is reduced it becomes more and more difficult to remove, and in a vacuum of 10^{-5} mm. Hg, 2 parts per million of oxygen by weight appears to be the lowest attainable. This condition is not improved much by raising the temperature from 1150° to 1300°C for instance, because the vapour pressure of copper increases and this again lowers the dissociation pressure.

Graphite crucibles have for some considerable time been used for casting copper in vacuum. Nevertheless, copper less than 2 ppm oxygen content has not been made commercially available by their use.

It has already been shown that gas continues to be emitted from graphite in vacuum at temperatures up to 2150°C., and that oxygen is readily re-absorbed on exposure to

[*Price 4s. 6d.*]

air. Oxygen can then only be removed at high temperature as carbon monoxide. Purity of graphite is important as it has been found that graphite of low ash content can be more completely de-gassed than that of higher ash content.

Thus for optimum oxygen removal from molten copper the following conditions must be met;—

(1) Low partial pressure of oxygen in vacuum system to prevent re-oxidation of copper to cuprous oxide.

(2) Low partial pressure of carbon monoxide to include cuprous oxide to reduce to metal.

(3) High temperature of reaction with graphite (approximately 1400°C) to give maximum free energy change and more complete oxide (and dissolved oxygen) removal. High temperature to cause rapid diffusion of oxygen through the molten copper to the surface.

(4) The graphite used for the reaction should be de-gassed to good vacuum (i.e. down to 1×10^{-5} mm. Hg or better) at as high a temperature as is practical (approximately 1800°C) and must remain continuously in that vacuum throughout the whole of the refining and casting process. Exposure to air would immediately contaminate the graphite. Nitrogen contaminates to a lesser degree.

(5) There should be maximum contact area between molten copper and de-gassed graphite to speed reduction of oxide.

(6) Molten copper should be broken down into thin streams and thin layers so that oxygen more quickly diffuses to the surface for the reaction to proceed.

The invention can be better understood from the following description given in relation to the accompanying drawing, in which:—

Fig. 1 is a view in vertical section of the refining tower complete with silica envelope and heating coils;

Fig. 2 is a view in plan of the circular wall of the refining tower, showing the type of construction.

Referring to Fig. 1, the apparatus consists of a graphite upper mould (A) from which copper or copper alloy is poured to a three section graphite refiner (C, D, E). The metal then pours into the lower graphite mould (H).

The whole assembly is mounted on a stem (W) and supported integrally by molybdenum pins (P). A transparent fused silica envelope (Z) surrounds the assembly leaving a small annular space around the assembly for vacuum transfer purposes.

The prevailing vacuum is induced by a vacuum diffusion pump backed by a two stage rotary pump.

Surrounding the silica envelope are situated the induction coils (X and Y) which are arranged in series and can be operated as separate units.

Before the start of a refining operation, all graphite parts of the assembly are furnaced at 1800°C to a vacuum better than 1×10^{-5} mm Hg and are then let down in pure nitrogen to atmospheric pressure when cold.

The upper mould (A) is then charged with high grade copper alone or together with the other high purity metal ingredients when refining copper alloys. The whole system is then evacuated to a pressure of 1×10^{-5}mm. Hg.

Lower mould (H) and refiner (C,D,E) are heated by induction to a temperature of 1800°C and maintained at this temperature until pressure falls to 1×10^{-5}mm.Hg or better. Metal held in upper mould (A) remains un-melted during this operation.

Induction coils (X and Y) are then rearranged and power input adjusted so that at the same time the upper mould (A) is heated to melting temperature, refiner (C,D,E) is maintained at 1400°C. and lower mould (H) is heated to 1150°C.

The copper or copper alloy ingredients then melt in the upper mould (A) and pour in a steady broad stream through the perforations (B) to the refiner (C,D,E) and fall over the graphite steps of the refiner. During this operation a vacuum of 6×10^{-6} mm. Hg is maintained external to the moulds and refiner. Molten metal, having been refined at 1400°C., enters the lower mould (H) and remains molten until the whole of the metal has passed from the upper mould and refiner.

Using only section (Y) of the induction coil, the copper or copper alloy is maintained in the molten state for half an hour to complete alloying and de-gassing. The heating coil is subsequently raised to heat the upper portion of the mould (H) to create a rising temperature gradient from bottom to top of mould. Horizontal isotherms are created and a shrinkage free casting is produced. When solidification is complete heating is discontinued.

When cold, vacuum is let down with pure nitrogen to atmospheric pressure and apparatus is ready for a repetition of the refining process.

The resultant ingot of pure copper or copper alloy has been shown to be extremely low in oxygen, nitrogen and hydrogen content and can be machined directly as contact material.

Fig. 2 of the accompanying drawing shows how the graphite moulds (A) and (H) and refiner (C,D,E) are provided with evenly

spaced slots in their outer surfaces, these slots extending from top to bottom of each part.

It has been shown thereby that efficiency of operation is greatly improved as the current induced by high frequency oscillation flows very superficially in the material heated. By slotting, the path length is greatly increased and results in greater resistance, hence better coupling results, and there are lower power losses between coil and material than would be obtained without the slots.

Additionally, moulds slotted in this manner can have a temperature on the inside surfaces some 150—200°C. higher than on the outside. Hot spots are created at the bases of the slots and these are closer to the inside surface than the outside so that radiation losses from the external surfaces are reduced considerably, particularly when working in the region of 1400° to 1800°C.

It follows that for a particular power output of an induction coil, a considerably higher temperature will be obtained by the use of slots in the walls of a mould. This results in a saving in power and a reduction in the size of equipment required to produce a given ingot cast, so reducing the cost of the contact materials.

This graphite refining tower operated entirely in vacuum has been designed to reduce the oxygen content of copper and dilute copper alloys (e.g. 1% Silver/Copper, 3—10% Antimony/Copper) to one part by weight of oxygen per ten million parts of copper or alloy. Nitrogen and hydrogen contents are at the same time reduced.

Copper produced by this process shows significant improvement as regards gas content over other qualities of copper commercially available and would be suitable for example for the production of contact materials for a high powered vacuum switch or circuit-breaker.

WHAT WE CLAIM IS:—

1. A refining tower for the removal of oxygen from copper and copper alloys, characterised in that the tower is constructed of graphite and is made with longitudinal slots in the outer surface of the graphite to increase the heating efficiency, these slots being spaced equidistantly round the tower, which is enclosed in a fused silica envelope, within which a high vacuum can be created by known mechanical means and characterised in that heating can be supplied from induction coils surrounding the silica envelope.

2. A refining tower according to Claim 1, characterised in that the tower comprises a graphite upper mould to which metal is charged in order to be melted, a graphite refiner through which molten metal from the upper mould can flow over an arrangement of steps, and a graphite lower mould which receives the molten metal after it has passed through the refiner.

3. A method of refining copper and copper alloys, which comprises charging solid metal to the upper mould of a refining tower as claimed in Claim 2, heating the mould until the metal is melted, and then refining the metal at 1400°C and a vacuum of 6×10^{-6} mm. Hg.

4. Copper and copper alloys whenever refined by means of a refining tower as claimed in Claim 1 and Claim 2.

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1052907 COMPLETE SPECIFICATION

1 SHEET This drawing is a reproduction of the Original on a reduced scale

FIG. 1.

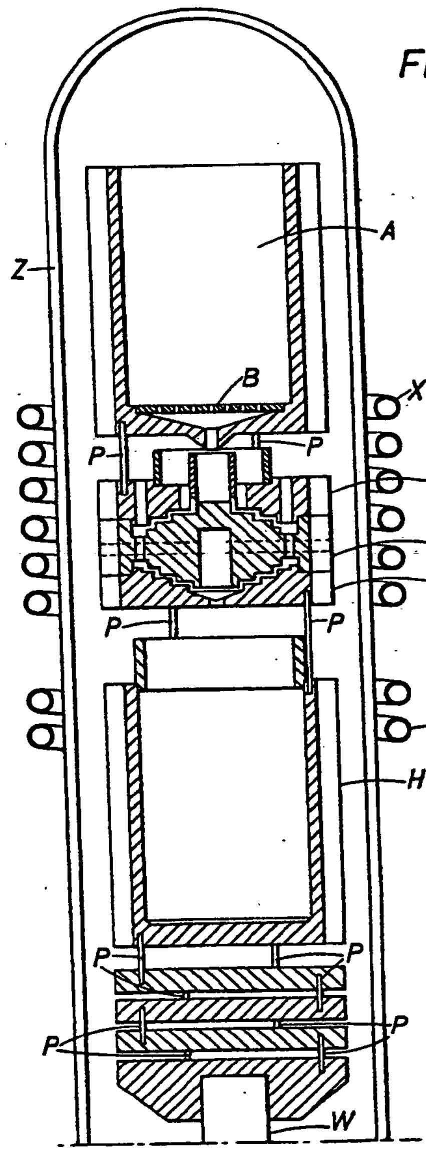

FIG. 2.